KARL O. HERZ and STEPHEN S. CHANG
Department of Food Science, Rutgers—The State University, New Brunswick, New Jersey 08903

An Apparatus for the Isolation of Volatile Compounds from Foods

SUMMARY

Volatile compounds that may contribute flavor to a wide variety of foods can be isolated in an apparatus which is described. It utilizes the principle of flash evaporation and vaporization from a continuous thin heated film. Liquid foods, such as fruit juices or beer, need no preparation, but foods such as meat and potato chips are first made into a fine water slurry. Vaporized food constituents and water are recovered by condensation in a series of specially designed cold traps. Features and performance of the apparatus are discussed.

INTRODUCTION

Of the steps that lead to knowledge of the chemical compounds that may contribute to the flavor of foods, identification procedures have seen enormous advances, primarily because of improvements in the design, operation, and performance of instruments for micro-separation of mixtures, such as the gas chromatograph, and for micro-identification of pure compounds, such as infrared, mass, and NMR spectrometers. By contrast, methods for the isolation of volatile trace flavor compounds have often had to be tailored to the requirements of the particular food under investigation; as one result, advances in design, operation, and performance of such apparatus have not been nearly so spectacular and have not approached the more universal usefulness of the procedures and instruments employed for identification.

Apparatus for the isolation and recovery of volatile constituents from oils without the creation of artifacts during isolation has been reported (Chang, 1961). This apparatus has served well in studies of soybean oil (Smouse and Chang, 1965), hydrogenated soybean oil (Kawada *et al.*, 1966), and cottonseed oil (Mookherjee and Chang, 1965). The present apparatus was designed to isolate volatile flavor compounds from beef. Its performance in this application points to a general usefulness with any food that is an aqueous solution or that can be dissolved or dispersed homogeneously in water. Experiments that have been conducted, though not reported here, have demonstrated this performance capability for such widely different foods as potato chips and beer. In the present apparatus, volatile substances are separated under reduced pressure by a combination of flash evaporation and vaporization from a thin heated film without significant creation of artifacts or decomposition of the food sample.

EXPERIMENTAL

Apparatus. The apparatus (Fig. 1) may be considered as consisting of three systems. The first system is designed to bring the liquid food or water slurry of solid food to conditions optimum for metering it into the second system, designed to separate trace volatile substances and water by vaporization under reduced pressure. In the third system, volatilized material is condensed.

The first system includes a 3-neck 5-L flask (*B*) which holds either the food or a fine water slurry of the food. The contents of the flask can be heated with a Glascol heating mantle and stirred with a precision-ground stirrer (K-78100, Kontes Glass Company, Vineland, N. J.). Significant loss of volatile substances to the atmosphere is prevented by a cold-finger trap (*A*) filled with dry ice. The slurry in flask *B* is pumped through capillary borosilicate tubing into the suction port of the positive-displacement rotating and reciprocating pump (*C*) (FMI Lab Pump, supplied by Heat Systems Company, Melville, New York).

The second system begins at the exit port of pump *C*, which is connected to the modified Alihn-type vaporizers (*D*) through a stainless-steel ball joint. The two vaporizers, connected in a series, are initially evacuated to about 0.01 mm Hg. Depending upon the rate of feed of slurry or liquid, the actual pressure during a run is considerably higher. The bulbs of the two vaporizers are bathed by a suitable circulating liquid heated to a temperature that will afford maximum assistance in vaporizing without risking decomposition of the food.

After the slurry leaves the exit port of pump *C*, there occurs a combination of flash evaporation and vaporization from the thin heated film passing down the walls of the bulbs. The portion of the

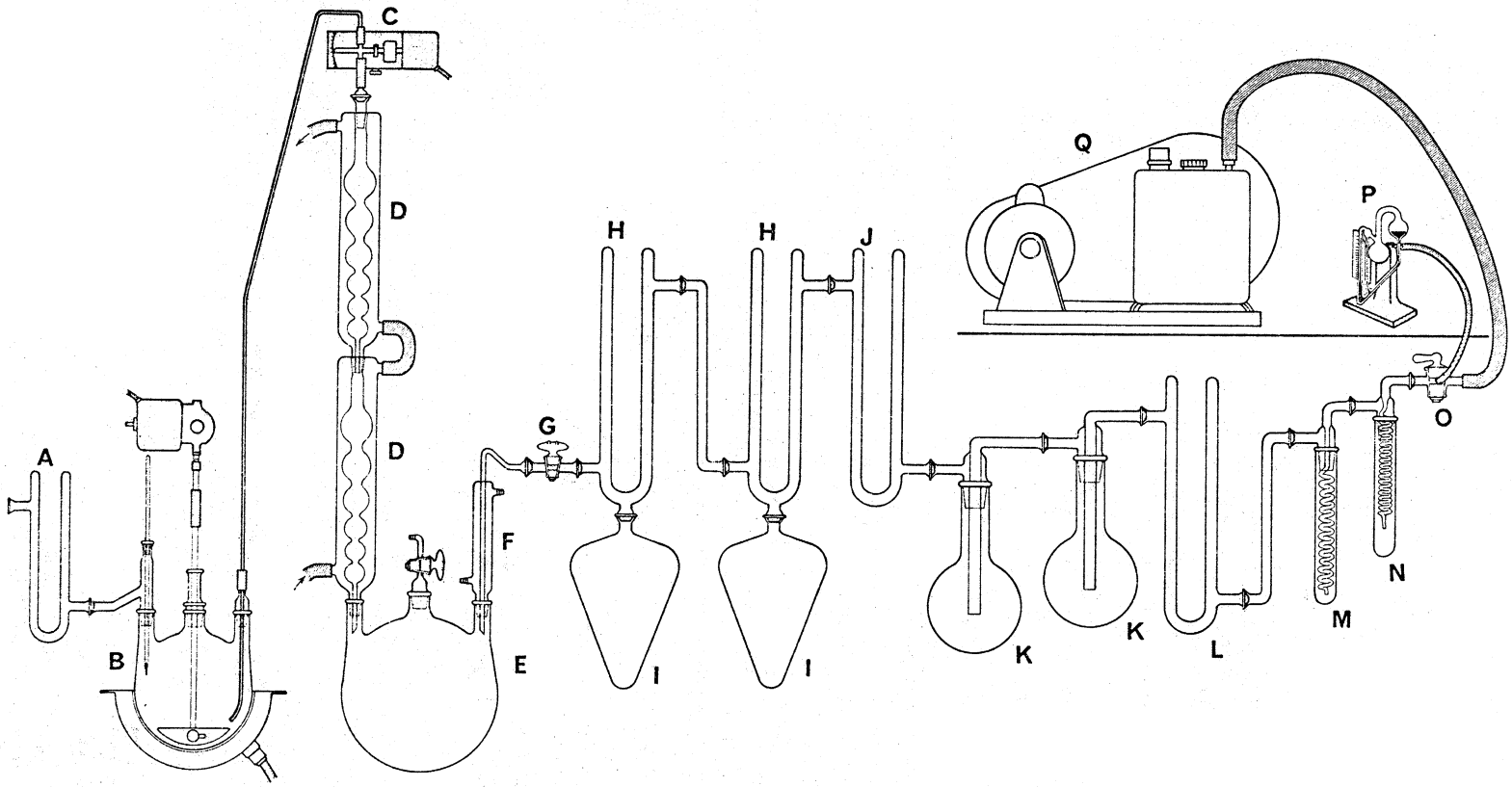

Fig. 1. Apparatus for the isolation of trace volatile constituents from foods.

food or beverage that is not volatile under these conditions drops into the 12-L round-bottom flask (*E*). The flask is normally not heated at the bottom, but mild heating of its top surface may assist in transferring vaporized material out of the flask. The exit tube (*F*) passes through a condenser jacket that may be utilized either for hot bath fluid (which assists the removal of the vaporized material toward the cold traps), or for cold liquid (which effects some slight preliminary cooling of very hot and rushing vapor).

The vaporizing system is separated from the condensing system by the stopcock (*G*), 15 mm bore, permitting the former to be maintained under reduced pressure while the latter is dismantled, cleaned, and reassembled for a second run. After reassembly, pressure in the vaporizing system may be reduced by evacuating through the adapter mounted in the center neck of flask *E*.

The kind of arrangement of cold traps employed in the condensing system may differ with the nature of the food and the conditions of vaporization. The arrangement shown in Fig. 1 has been found suitable for use with a water slurry of boiled meat. The first two dry-ice-cooled traps (*H*) and the 4-L conical receiving vessels (*I*) serve to condense the bulk of the vapors. Conditions can be found without difficulty which will result in condensation of vapor to liquid on the walls of the cold fingers of traps *H*; the condensed liquid drips off the tip of the cold finger into the conical flask *I*, which is set in a pail containing dry ice. Cold-finger trap *J* may be replaced by another set of the *H-I* combination if additional capacity is desired. The volume of liquid collecting in the different receiving vessels can be controlled by varying the level of fill and the frequency and intensity of stoking of the dry ice in the cold fingers above them. The remaining cold traps (*J* and *K*) are cooled by dry ice, and traps *L*, *M*, and *N* by liquid nitrogen.

The specially designed glass parts of the apparatus can be obtained from either Kontes Glass Co., Vineland, N. J., or from Scientific Glass Apparatus Co., Bloomfield, N. J.

Procedure. The isolation of volatile flavor compounds from boiled beef is used to illustrate this apparatus. With certain modifications of procedure, the apparatus has been similarly used for the isolation of flavor compounds from potato chips and from beer.

Cold boiled meat, 1 kg, was ground in a meat grinder (⅛-inch plate), and subsequently made into a fine slurry by comminuting in a Waring blender with just enough cold water (3400 ml), to permit effective cutting action of the blades. The flavor characteristics of the meat were found not to be altered by the grinding and blending. The fine slurry was held in flask *B* for 1 hr under moderate agitation at 70–75°C in order to develop the desirable flavor. During this time, the vaporization and condensation systems of the apparatus were evacuated to less than .03 mm Hg.

Pump *C* was then started to feed the slurry at a relatively rapid rate (80 ml/min) into the vaporizer units (*D*), the bulbs of which were bathed in circulating glycerine kept at 105–110°C. The temperatures of slurry and vapor inside flask *E* were respectively 28°C and 50°C. A typical pressure reading above the slurry was 15 mm Hg, and that at the end of the cold traps was 3 mm. A total of 710 ml of condensate was collected in the cold traps. The condensate had a strong true boiled-beef aroma. The condensate in the two coiled traps cooled with liquid nitrogen had a strong pungent odor suggestive of sulfur compounds.

To ensure as complete as possible a removal of unaltered volatile flavor compounds from the beef slurry, the isolation process was repeated. Stopcock *G* was closed, and vacuum in flask *E* was released. The slurry residue was returned from Flask *E* to *B*, reheated to 70–75°C, and held at this temperature for 1 hr. Meanwhile, the vaporization system was evacuated through the adapter fitted in the center neck of flask *E*. Stopcock *G* was then opened, and the slurry was fed into the vaporizers at a rate of 35 ml/min. A total of 590 ml of condensate was collected. Since it still had a strong true boiled-beef aroma, the slurry residue was again submitted to the isolation process at a rate of 50 ml/min. The condensate collected the third time, 520 ml, had only a weak aroma.

The total condensate may be extracted with ethyl ether, and the ether extract concentrated according to the method of Chang (1961). The concentrated ether solution can then be fractionated by gas chromatography and the pure gas chromatographic fractions identified by infrared, and mass spectrometry. Exceptionally large compounds may yield fractions sufficient for micro NMR analysis. The volatile flavor compounds isolated from 56 lb of boiled lean beef were found sufficient for a systematic study of their gas chromatographic fractions by infrared and mass spectra.

DISCUSSION

The apparatus has been used successfully for the isolation of volatile flavor compounds from foods as different as meat, potato chips, and beer. The volatile compounds isolated from each food had a strong flavor characteristic of that food as evaluated organoleptically. Furthermore the intensity of the flavor of the food after it had been processed was greatly reduced. The apparatus is therefore

flexible and may have a wide range of potential applications.

Conditions that must be determined experimentally for each food include the temperature and time of holding for flavor development, the rate of metering into the vaporizers, and the temperature of the liquid circulated through the vaporizer jacket. As the slurry enters the evacuated system, flash evaporation occurs, and the portion of the slurry that is not vaporized undergoes an instantaneous considerable drop in temperature. The effect of this drop is substantially mitigated as the slurry travels along the heated vaporizer bulb walls in a thin, continuous film. Bubbling due to vaporization from the film occurs throughout the length of the vaporizers.

Conditions must be chosen so that the largest possible quantity of flavor compounds is stripped from the food without causing alteration of the food with respect to flavor and without the creation of artifacts. In some cases, for more complete isolation of the volatile flavor compounds, the food may pass through the apparatus more than once.

Two difficult problems encountered in developing the apparatus were selection of a metering pump and design of the cold traps. The pump must be able to feed the food from a flask under atmospheric pressure into a system under high vacuum. The positive-displacement reversible pump operating on the rotating and reciprocating piston principle serves the purpose well. The train of cold traps must accomplish condensation of both the relatively large volume of water vapor and the low-boiling flavor compounds volatilized in the vaporizers. Most of the water vapor is effectively removed by condensation to the liquid state on the cold-finger traps. The liquid condensate drips off the cold finger into a conical receiving flask. The conical shape is necessary to prevent breakage when the liquid freezes. The remaining cold traps are more conventional design for condensation of the vapor to solid state. Liquid nitrogen is used as coolant for the last three traps to ensure virtually complete trapping of condensable flavor compounds.

REFERENCES

Chang, S. S. 1961. A new technique for the isolation of flavor components from fats and oils. *J. Am. Oil Chemists' Soc.* **38**, 669.

Kawada, T., B. D. Mookherjee, and S. S. Chang. 1966. Chemical reactions involved in the catalytic hydrogenation of oils. III. Further identification of volatile by-products. *J. Am. Oil Chemists' Soc.* **43**, 237.

Mookherjee, B. D., and S. S. Chang. 1965. A chemical study of the volatile decomposition products of a slightly autoxidized cottonseed oil. Presented at Meeting of Am. Oil Chemists' Soc., Cincinnati, Ohio.

Smouse, T. H., and S. S. Chang. 1965. A systematic characterization of the reversion flavor of soybean oil. Presented at Meeting of Am. Oil Chemists' Soc., Houston, Texas.

Ms. rec'd 3/29/66.

Supported by Agricultural Research Service, U. S. Department of Agriculture Grant No. 12-14-100-7669 (73), administered by the Eastern Utilization Research and Development Division, Philadelphia, Pa.

Paper of the Journal Series, New Jersey Agricultural Experiment Station, Rutgers, The State University of New Jersey, Department of Food Science, New Brunswick.